TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento
Corrective Action Report

Title: SSAS13-3-533 320-J4473

Reference: 58-39

Submitted by: Thep Phomsopha **Date: 11/25/13**

Mercury in Impinger
Method 29 (CVAA)

Parameter	Reported Value	Assigned Value	Acceptance Range
Mercury	159	15.0	11.3 – 18.8 ng/mL

Investigation Planned or Completed:
The raw data was reviewed for calculation and transcription errors, and for unacceptable quality controls in the calibration and fortified sample recoveries (i.e., ICV, ICB and laboratory check samples (LCS). No calculation or transcription errors were discovered. The initial and continuing calibration standards were within method criteria. All continuing calibration blanks were below the detection limit, as was the method blank prepared with the sample. All quality controls were acceptable.

The procedure for documenting the preparation of PT samples was reviewed for proper documentation as established when PT samples are analyzed. The analyst included the PT instructions and the PT Sample Prep Form in the sample preparation section associated with the sample.

Root Cause Analysis
Samples for Method 29 analyzed for mercury are routinely analyzed with an initial volume of 3mL and a final volume of 30mL, which constitutes a 10X dilution. This is done to reduce the concentration of hydrogen peroxide in the samples. When the PT Audit sample was batched a 3mL initial volume was inserted in the field, however, the sample was actually prepared for analysis using a 30mL initial volume. The secondary review analyst would not have been able to verify the actual prep volume for mercury analysis because the aliquot used for analysis did not get recorded on the PT Sample Prep Form. Preparation of PT samples deviates from normal sample preparation procedures and a weakness in the process has been identified.

This error occurred because the Analyst used a fill down function when putting in the initial volume and forgot to change the Audit sample to a 30mL initial volume to reflect what was actually performed.

Corrective Action Plan
An immediate corrective action was to re-digest, and re-analyze the sample at a 1X and 3X dilution. The analytical results were within the Acceptance Range at 15.8 ng/mL.

The long term corrective action is to include the aliquot of sample used for preparation (Initial volume) on the PT Sample Prep Form so that the secondary review person has this information available to verify the correct sample preparation volume. This corrective action is effective immediately for processing PT samples.

The dilution error is specific to this PT sample. Analytical results for samples analyzed in the same batch as the PT sample are not impacted by the dilution error.

Closed by: _____ 12-12-13
Gary Costley Date
Quality Assurance Scientist

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Corrective Action Report

Title: SSAS13-3-533 320-J4473

Reference: 58-38

Submitted by: Thep Phomsopha **Date: 11/25/13**

Metals in Impinger
Method 29 (ICP-MS)

Parameter	Reported Value	Assigned Value	Acceptance Range
Cadmium	0.181	1.31	1.05 – 1.57 µg/mL
Lead	0.208	0.988	0.741 – 1.24 µg/mL

Investigation Planned or Completed:
The raw data was reviewed for calculation and transcription errors, and for unacceptable quality controls in the calibration and fortified sample recoveries (i.e., ICV, ICB, CRI, ICSA, ICSAB, Linear Range Standard and laboratory check samples (LCS)). No calculation or transcription errors were discovered. The initial and continuing calibration standards were within method criteria. All continuing calibration blanks were below the detection limit, as was the method blank prepared with the sample. All quality controls were acceptable.

The sample was originally analyzed at a 1X, 5X, and a 10X dilution and all results were outside the Acceptance Range. The results appear to be off by a factor of 10.

Evaluating the procedure for documenting dilutions was also investigated. Dilutions are normally prepared directly in the tubes to be loaded onto the auto sampler and the dilution is written on the tube. With this PT sample, a 10x dilution was first prepared in a small container based on high levels of silver (Ag) historically seen in the PT sample. The analyst was then momentarily distracted when his assistance was requested for another task. When the analyst resumed preparing the PT sample, tubes were labeled for dilutions at 5X, and 10X for analysis. The analyst forgot about the original 10X dilution as the first step in the preparation of the PT sample and further dilutions propagated the error and the tubes were incorrectly labeled.

Root Cause Analysis

- Preparation of the PT sample at a 10x dilution in a small container is different than the normal procedure of preparing dilutions for samples.
- Distracting the analyst at a critical time of sample preparation caused the analyst to lose focus at the task at hand.

Corrective Action Plan
Immediate corrective action was to re-digest and re-analyze the sample. The samples were analyzed at 5X and 25X dilution and the analytical results were 1.05 ug/mL (Pb) and 1.33 ug/mL (Cd); both within the established Acceptance range.

This appears to be an anomaly that does not routinely occur when the analytical sequence is being created with sample dilutions. The lesson learned is to minimize distractions when dilutions are being performed.

The dilution error is specific to this PT sample. Analytical results for samples analyzed in the same batch as the PT sample are not impacted by the dilution error.

Closed by: _____ 12-12-13

Gary Costley
Quality Assurance Scientist

Date